

High-Energy X-ray Enhancement Possibilities from an APS Upgrade

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High-Energy X-rays

Working definition ↙ Photons between 35 - 200 keV
50 - 120 keV

■ Low Absorption

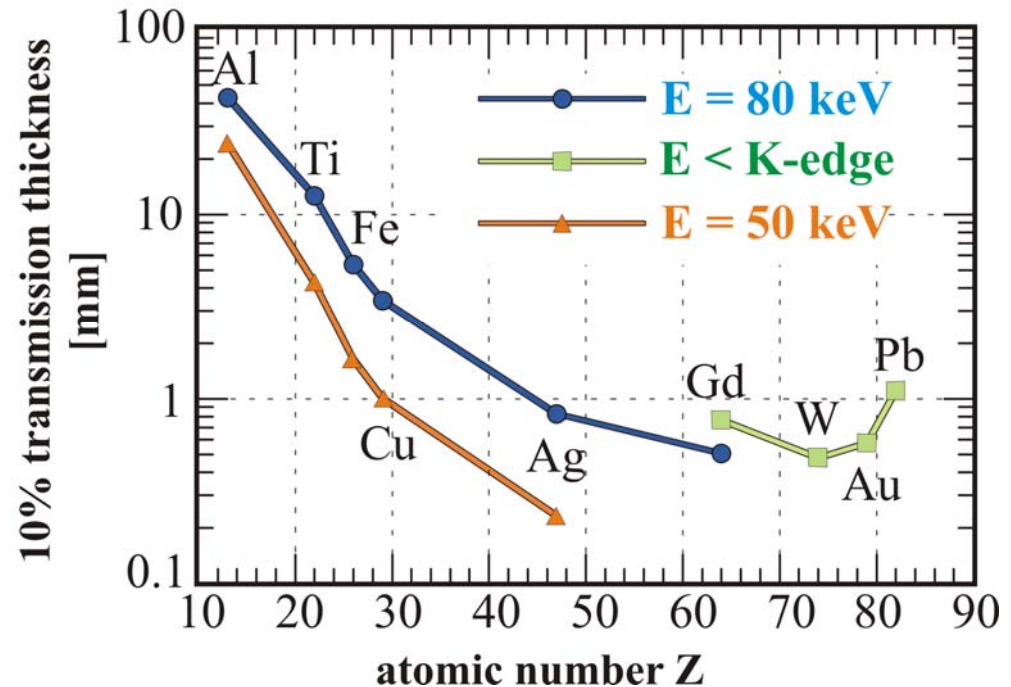
- Bulk measurements
- Special environments
 - *Furnaces*
 - *Reaction cells*
 - *Cryostats*
 - *High-pressure cells*
- Often comparable to neutrons

■ Simplified Scattering Processes

- Kinematical diffraction
- Small absorption, polarization, & dispersion corrections

■ Small Diffraction Angles

- Large Q range



Applications of High-Energy X-rays

Stress/strain/texture measurements

Small-angle scattering

3-Dimensional x-ray diffraction microscopy (3DXRM) (i.e., grain tracking)

PDF

Includes high-pressure

Powder diffraction

High-resolution (point counting)

Time-resolved (area detectors)

Diffuse scattering

Triple-axis diffractometry

Fluorescence measurements

Imaging

Tomography

Radiography

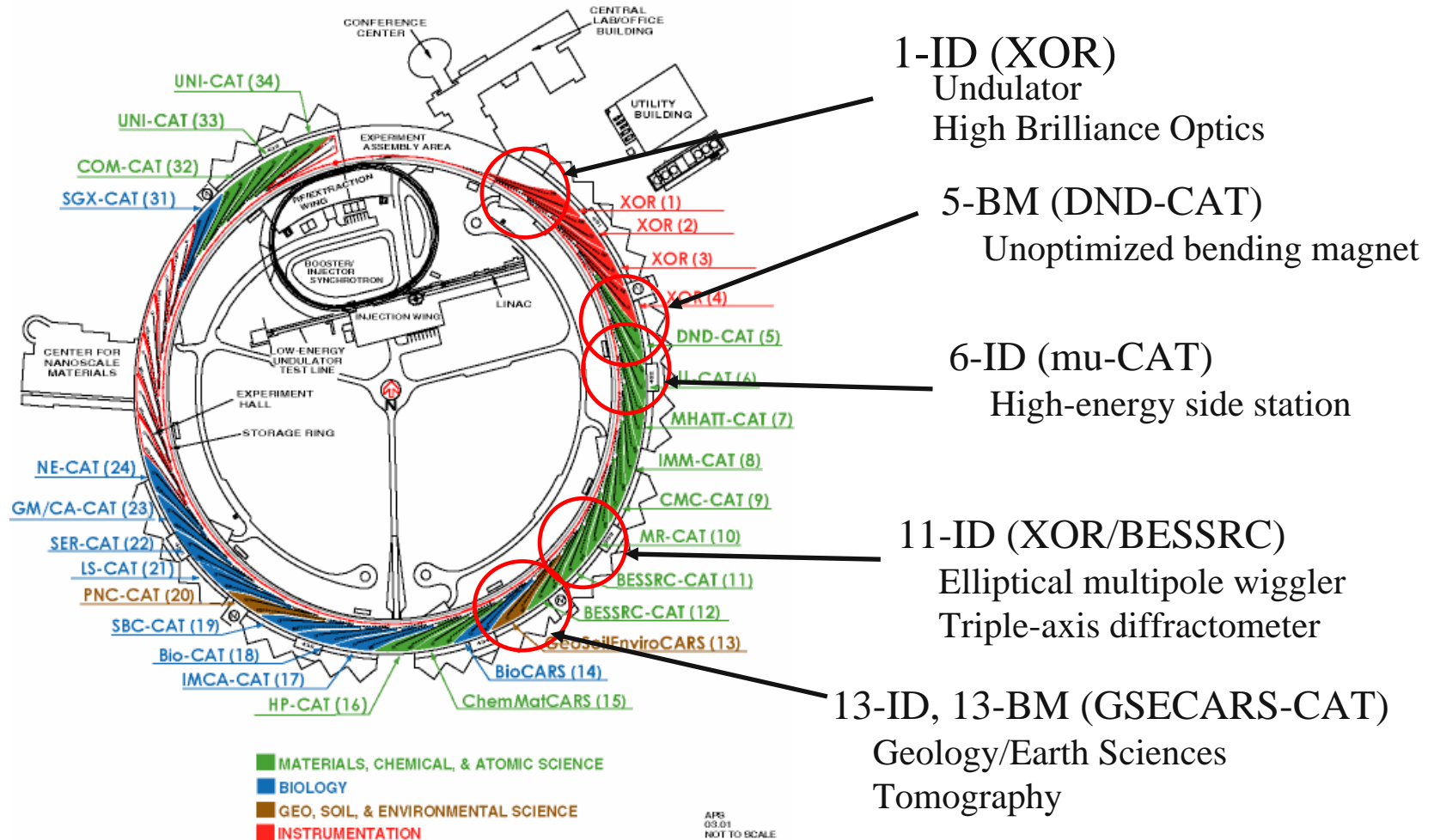
Combinations of the above

SAXS/WAXS

Imaging/WAXS

APS High Energy X-ray Facilities

APS Collaborative Access Teams by Sector & Discipline



1-ID Capabilities

As of October 4, 2005:

- Full time high-energy x-ray operation

- Upgrade underway, Phase I nearly complete

Optimized for high-brilliance, high-energy x-rays

- Typically 45 - 120 keV, fully tunable

- Focusing to < 10 microns

- Stability

Heavily oversubscribed (often 5X) for many years

Experimental scope

- Stress/strain/texture measurements

- HESAXS/WAXS

- 3DXRM (i.e., grain tracking)

- PDF (including high-pressure)

- High-resolution powder diffraction

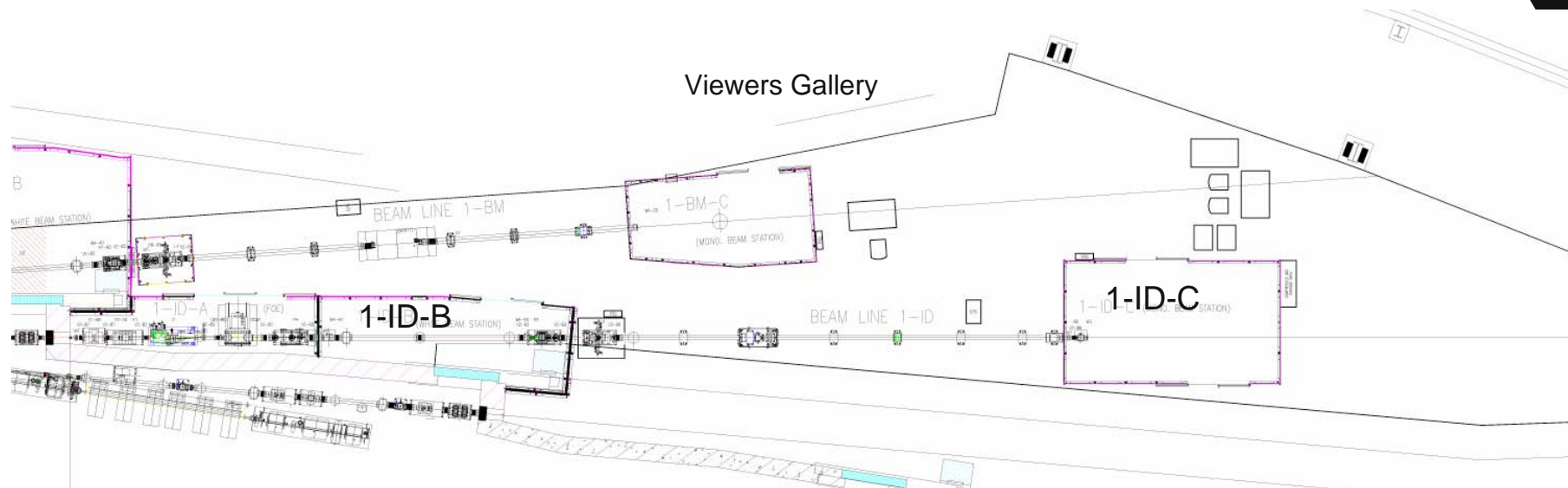
- Time-resolved (low-resolution) powder diffraction

- Diffuse scattering

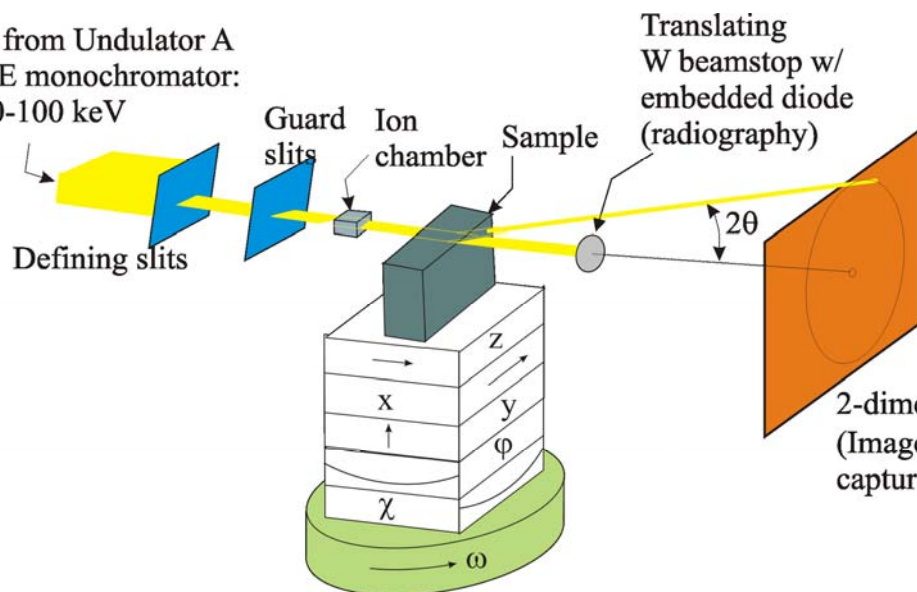
- Fluorescence measurements

- Optics development

1-ID Layout



Beam from Undulator A
and HE monochromator:
 $E \sim 50\text{-}100\text{ keV}$

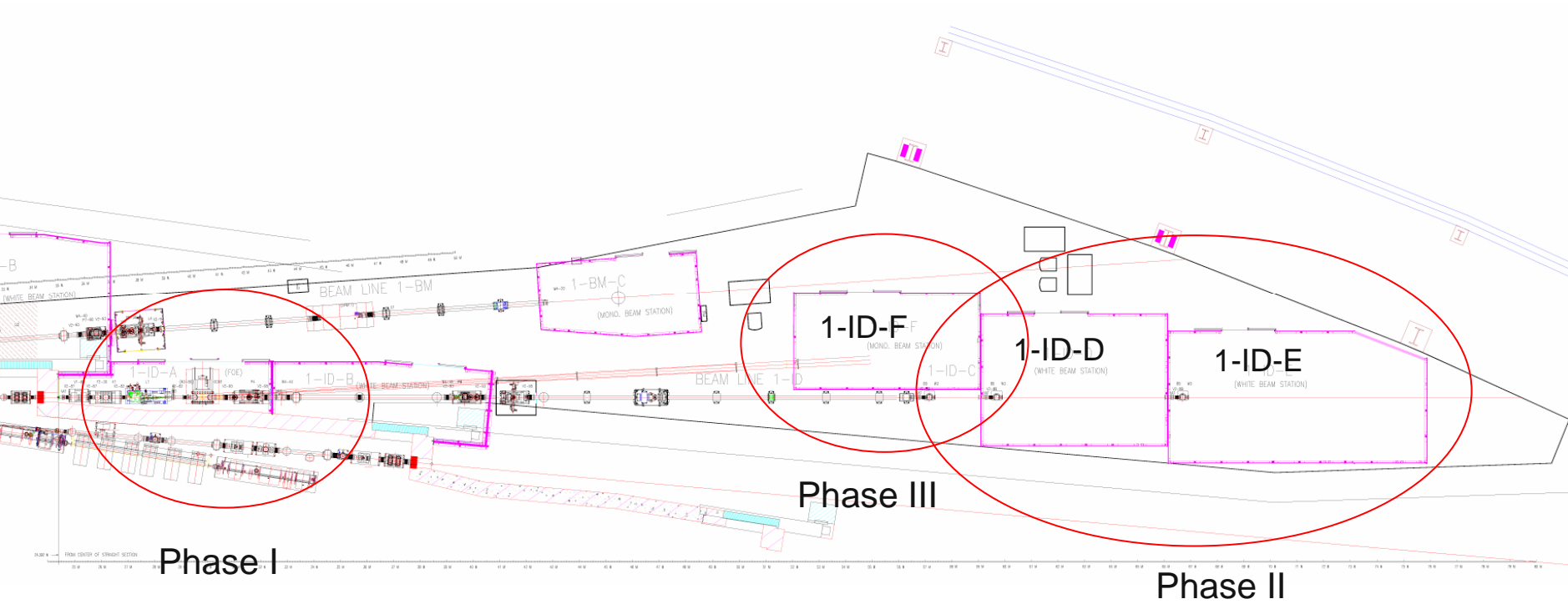


Features

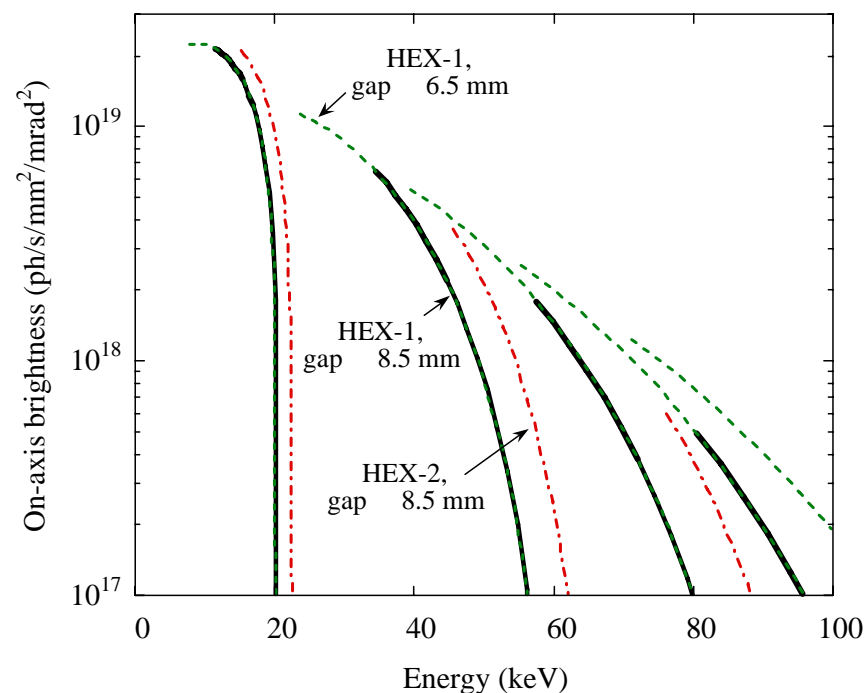
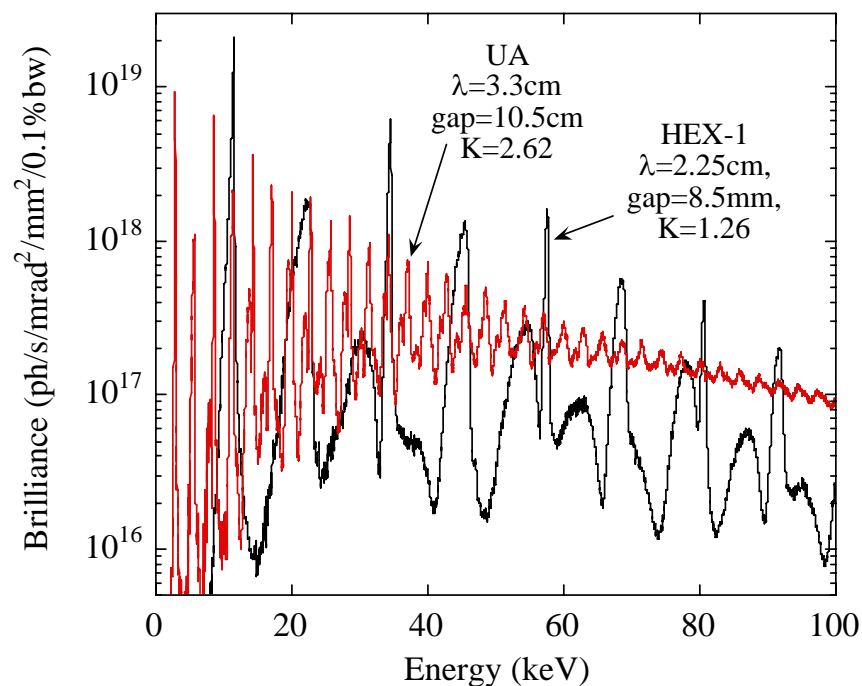
- $1 \times 2\text{ mm}^2 > \text{Beam size} > 0.01 \times 0.01\text{ mm}^2$ (with no focusing)
- Perform WAXS and radiography simultaneously
- Speed is highly dependent on detector

2-dimensional detector
(Image Plate $3.4 \times 3.4\text{ k}$, $100\mu\text{m}$ pixels):
capture full Debye cones

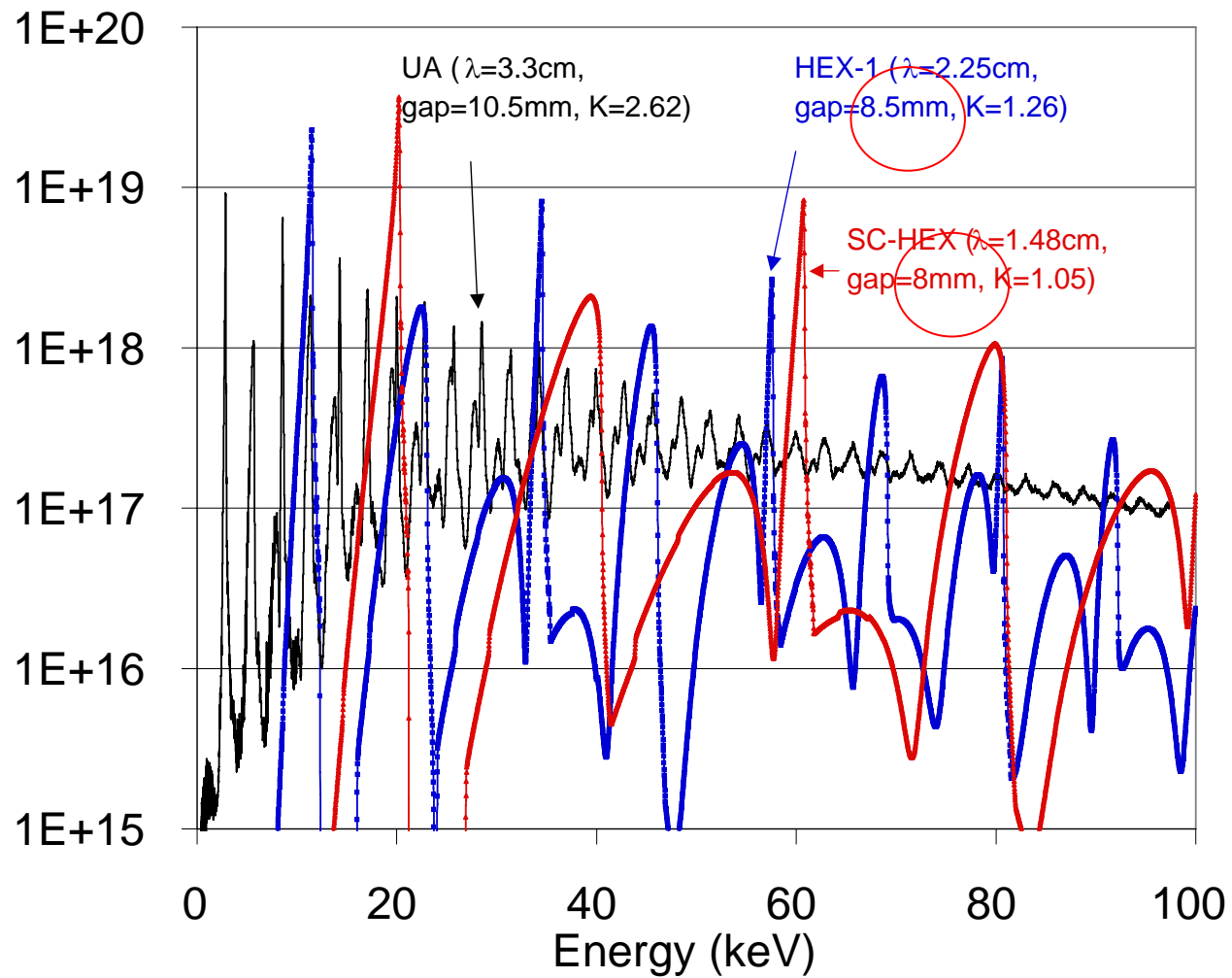
1-ID Upgrade Plan



Performance of a Specialized High-Energy Undulators



	UA	HEX-1	HEX-2
Period (cm)	3.3	2.25	2.05
Length (m)	2.5	2.5	2.5
Number of Periods	72	111	122
Minimum gap (mm)	10.5	8.5 (6.5)	8.5
B_{max} (T)	0.85	0.6 (0.85)	0.5
K_{max}	2.62	1.26 (1.8)	1.0
Total Integrated Power (kW)	5.4	2.8 (5.7)	2.1
Integrated Power in 1 x 1 mm ² @ 30 m (W)	160	168 (234)	155



11-ID Capabilities

Currently: Elliptical multi-pole wiggler, soon to upgrade to undulators

Three experimental stations

11-ID-D: 4-50 keV

EXAFS, XANES, time-resolved spectroscopy

11-ID-B, fixed energies 60-95 keV:

Magnetic Compton scattering (rarely used)

PDF

11-ID-C, fixed energies 60-115 keV:

High-energy diffractometer

Magnetic scattering

Diffuse scattering

Scope

Moving towards optimization for high-flux

Lots of PDF (mostly for nanoscience, chemistry)

Limiting scope due to staffing issues

Currently oversubscribed roughly a factor of 2, but growing

11-ID Layout

Toroidal &
Plan Mirror
Combination

11-ID-B: Beam penetrates 11-ID-D monochromator
(scattering plane is in ring-plane)

Offset
(vertical)

11-ID-C: Beam penetrates 11-ID-D & 11-ID-C monochromator
energy should be larger than 11-ID-C
(scattering plane is in ring-plane)

Inplane

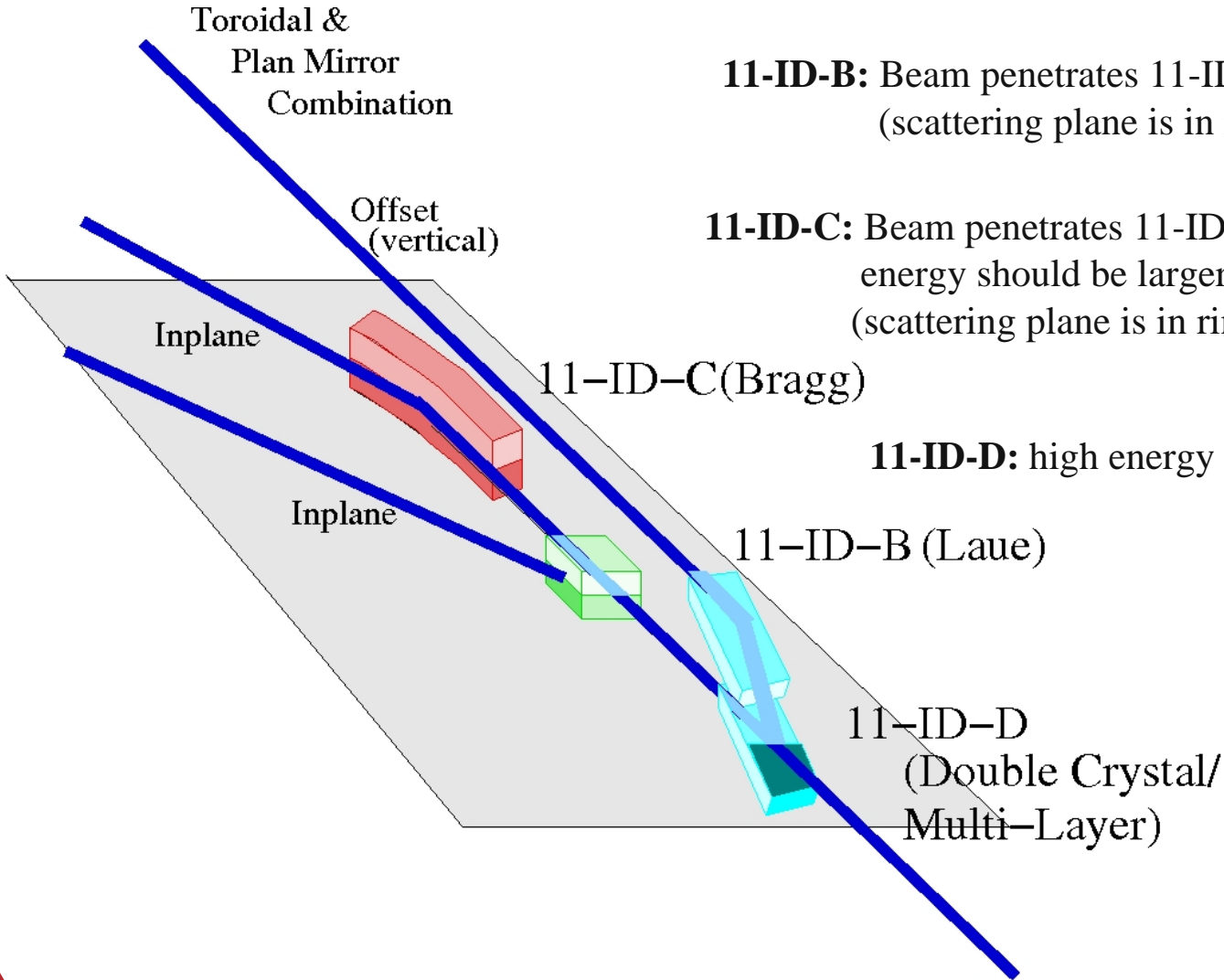
11-ID-C(Bragg)

Inplane

11-ID-D: high energy part penetrates first crystal

11-ID-B (Laue)

11-ID-D
(Double Crystal/
Multi-Layer)



Recent Workshops

- Workshop on High Energy X-rays Scattering at the APS (Argonne, Mar. 8 & 9, 2001)
 - APS Facilities
 - *Plans for HEX-CAT presented*
 - Crystallography (Coppens, Kramer, Wilkinson, Harlow, Argyriou, Petkov)
 - Stress/Strain/Texture (Withers, Dunand, Lienert, Marguiles, Ustundag, X. Wang)
 - General (Beckmann, Schneider, Honkimäki, Z. Zhong, Stock, Stremper)
- Workshop on Science with High-Energy X-rays (Argonne, Aug. 9 & 10, 2004)
 - Two days, parallel sessions on second day
 - 28 speakers, wide range of science
- ANLBESUF 2006 Users Meeting--Texture and Strain Mapping with X-rays, Neutrons, and Electrons (May 4, 2006)
 - About 1/2 talks had high-energy x-ray work
- Petra III/GKSS Workshop: High Energy Beamline at DESY (Hamburg, Germany, June 30, 2006)
 - Shastri, Haeffner from the APS
- New Structural Science from Improved High Energy X-ray Sources (Argonne, July 13, 2006)
- New Applied Materials Research from Improved High Energy X-ray Sources (Argonne, July 28, 2006)

New Structural Science from Improved High Energy X-ray Sources

■ July 13, 2006, 2:00 pm - 5:00 pm

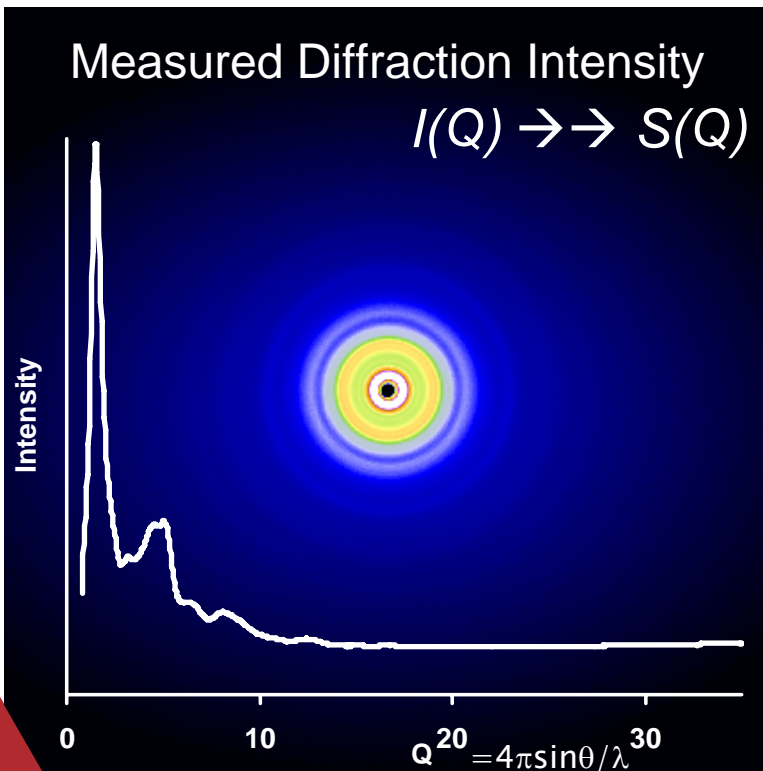
■ Coordinators: B.H. Toby, D. R. Haeffner, P. Chupas

■ Talks

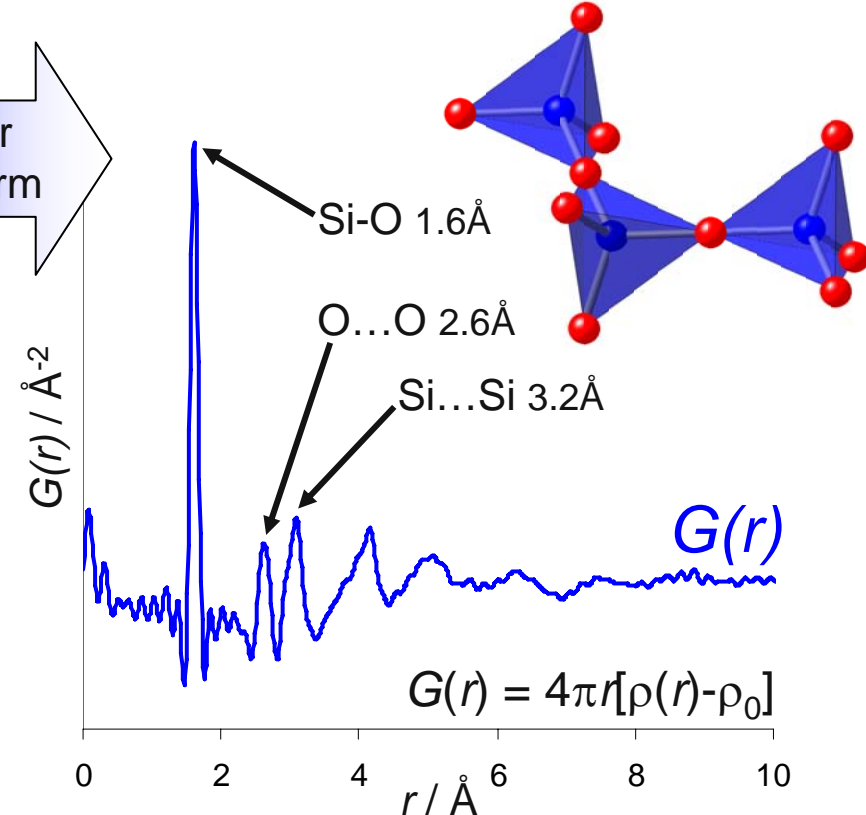
- Introduction—Dean Haeffner (APS)
- Application of PDF Measurements to Spatially Resolved and Time Resolved Measurements—Karena Chapman (APS)
- Opportunities for New High Pressure Science using High Energy X-ray Beams—John Parise (SUNY-Stony Brook)
- Resonant Scattering Employing High Energy X-Rays—Peter Lee (APS)
- Scattering Studies of Framework Materials At High Pressure—Joe Hriljac (U. of Birmingham, U.K.)
- Application of High Energy X-rays to Structural Studies ion Magnetic Fields—Yang Ren (APS)
- Discussion

The Pair Distribution Function Method

- Related to the *probability* of finding two atoms at a distance r
- For glasses, liquids, amorphous, nanocrystalline, heterogeneous, crystalline materials
- Gives coordination #'s, atomic distances, ~particle size, structural modeling



Fourier Transform



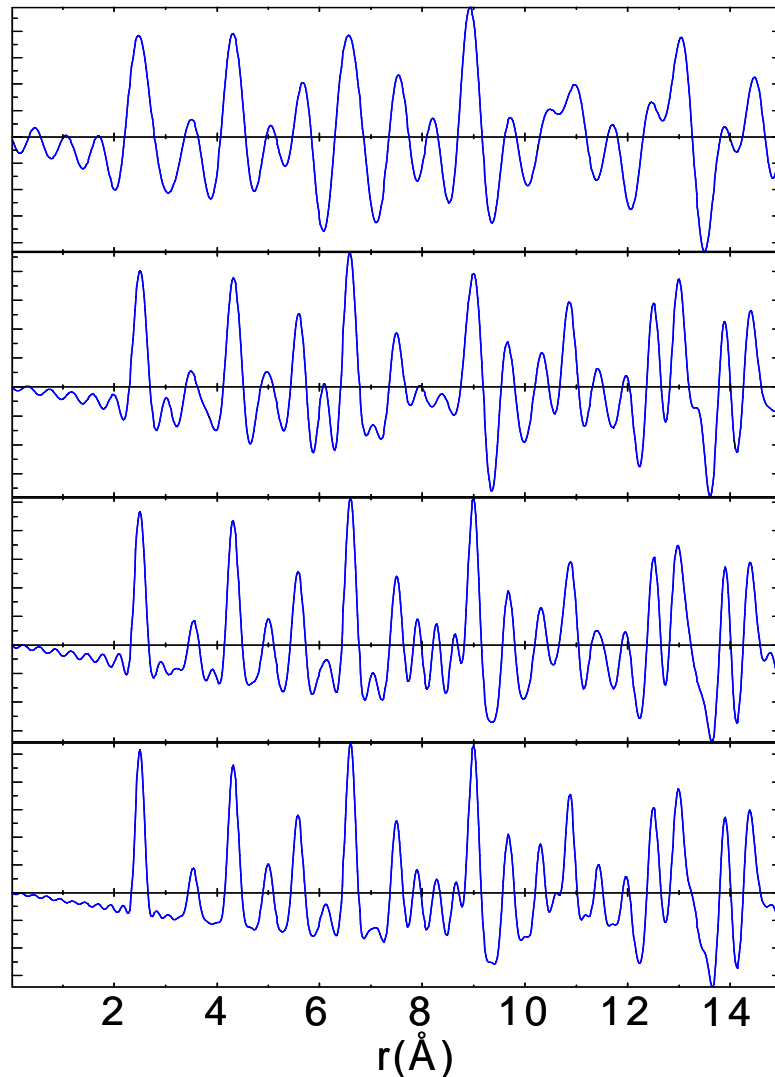
High Real Space Resolution PDFs: The Need for High Q Measurements

$$Q_{max} = 4\pi \sin\theta / \lambda$$

for Cu $K\alpha$, $\lambda = 1.54 \text{ \AA}$, $2\theta = 180^\circ$

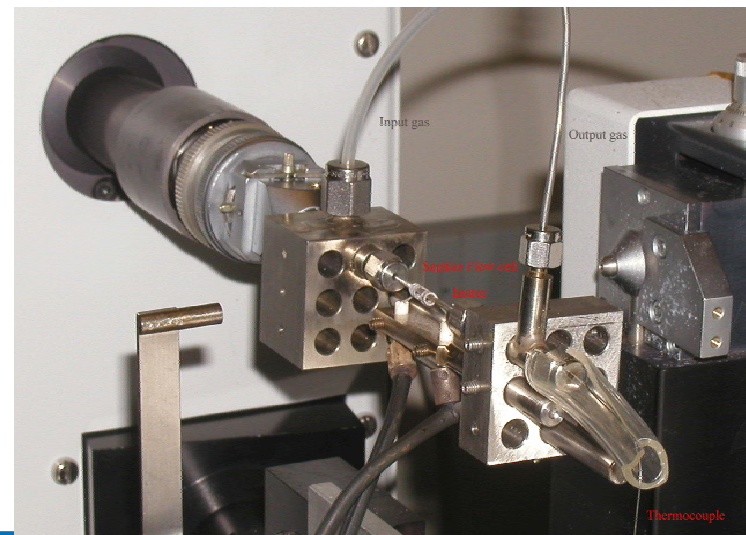
$$Q_{max} = 4\pi \sin 90 / 1.54 = \underline{8 \text{ \AA}^{-1}}$$

Need *Larger Detectors*
Coverage
and *shorter wavelengths*



The Application of Area Detectors to PDF Measurements

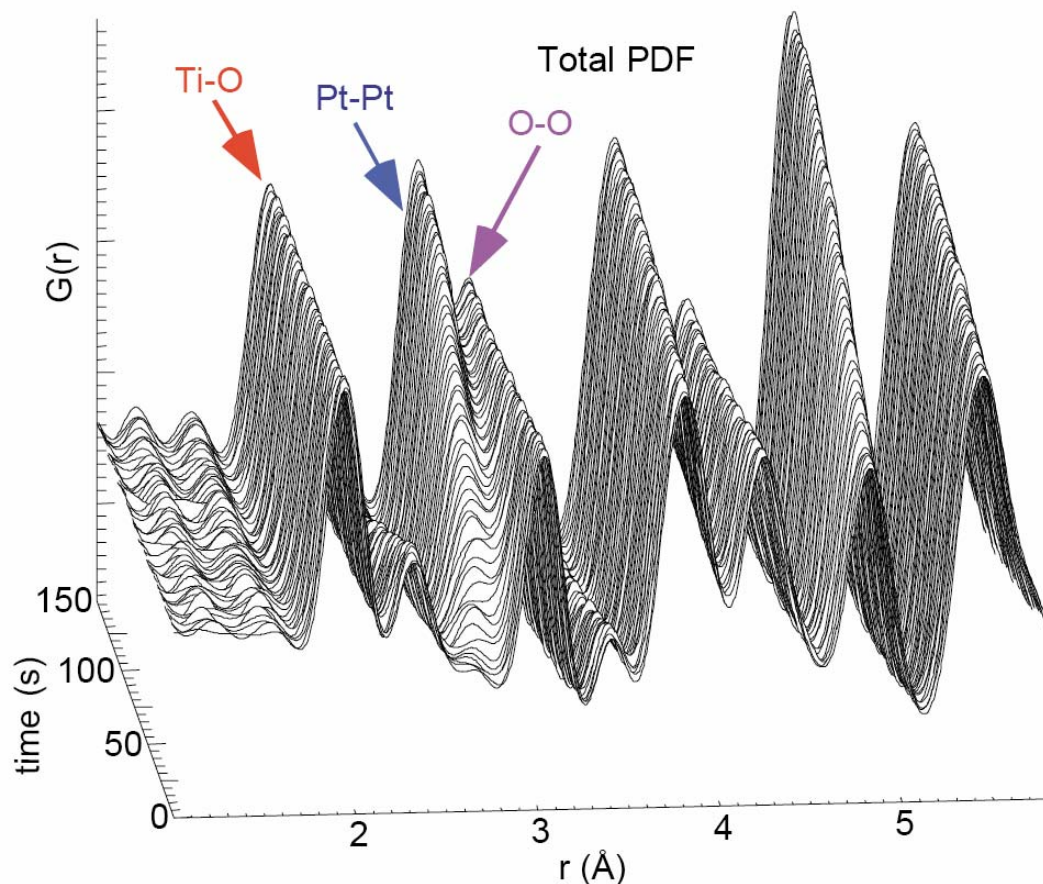
- Conventional Measurements
 - scanning point-by-point data collection
 - relatively slow – 12-24 hr / PDF
- Since 2002
 - area detectors - imaging plate
 - simultaneously measures scattering intensity to high Q
 - Image plates faster but still readout ~2min.
 - enable parametric and *in-situ* studies
- Recent Developments (nb. APS Detector Pool)
 - GE amorphous Si
 - Readout as fast as ~30ms



Time-Resolved Studies of Chemical Processes:

Understanding Catalysis In-Situ

The reduction of Pt^{4+} supported on TiO_2 to Pt^0 under H_2 at 200 °C



- The data shown were collected at a rate of 0.5 second/PDF.

- Data collection at a 33 millisecond rate (the fastest allowed by the GE a-Si detector) has been achieved.

P.J. Chupas, K.W. Chapman,
C.P. Grey, P.L. Lee

Consensus Comments

- More flux on sample
 - Not necessarily brilliance
- Detectors - fast 2D
- Detectors - fast 2D
- Detectors - fast 2D
- Sample environments
 - Dedicated to a station

New Applied Materials Research from Improved High Energy X-ray Sources

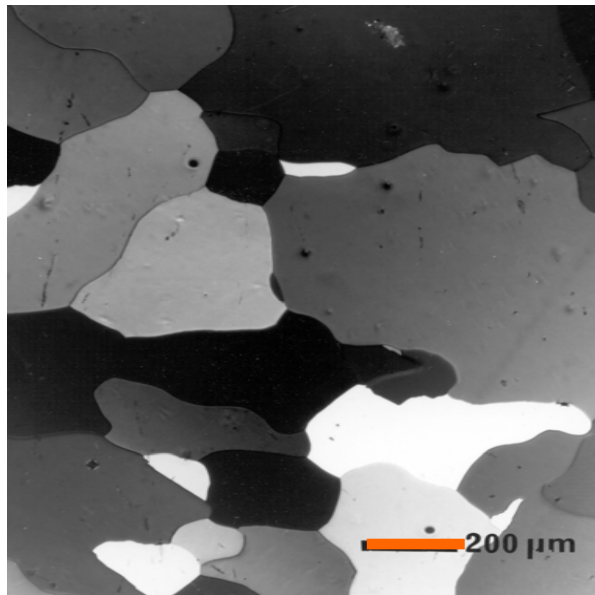
■ July 28, 2006, 9:00 am - 12:00 pm

■ Coordinators: B.H. Toby, D. R. Haeffner, P. Chupas

■ Talks

- Introduction and Recap of Recent Workshops–Dean Haeffner (APS)
- High-Energy X-Ray Optics and the APS Upgrade–Sarvjit Shastri (APS)
- High-Energy SAXS/WAXS for Materials Research–Jon Almer (APS)
- Opportunities for 3DXRD at the APS–Ulrich Lienert (APS)
- New Possibilities for Strain Pole Figures–Joel Bernier (APS)
- Discussion and Wrap Up

- metals, ceramics (structural, functional)
- *In situ* structural characterization during thermo-mechanical processing
- Surface not representative => High energies
- Micro-mechanical coupling of heterogeneous grains
- Length scales:
 - Defects: dislocations, precipitations (nm – μm) micro (EM)
 - grains: (nm – mm) meso
 - components: (> mm) macro (neutrons)

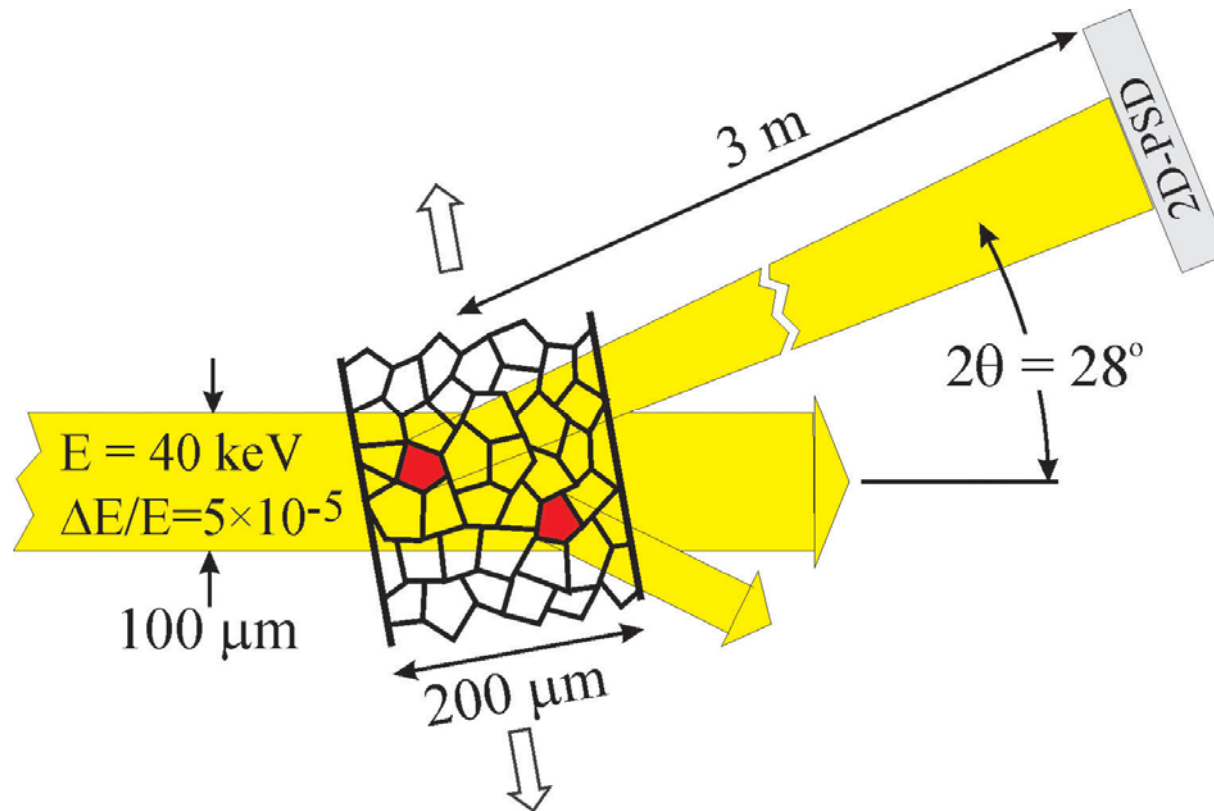


- Crystallographic phases: 1D
- Real space: 3D
- Orientation: 3D
- Strain: 6D
- Time: 1D
- Mapping of 14D parameter space
- Area detectors:
 - significant data acquisition rate
 - 'tomographic' diffraction: reconstruction software

Mesoscale Grain-Grain Interactions

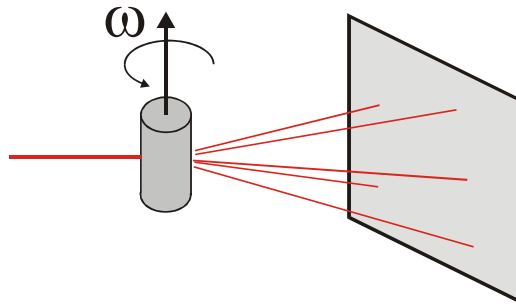
U. Lienert, J. Almer (ANL), T. Leffers, L. Margulies, S. Nielsen, W. Pantleon, H.F. Poulsen, and S. Schmidt (*Risø National Lab, Denmark*)

Experimental Setup

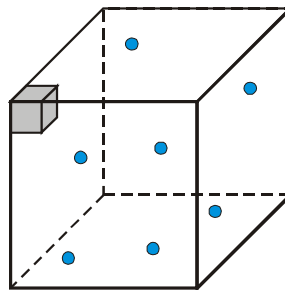


Grain Orientation: *GRAINDEX*

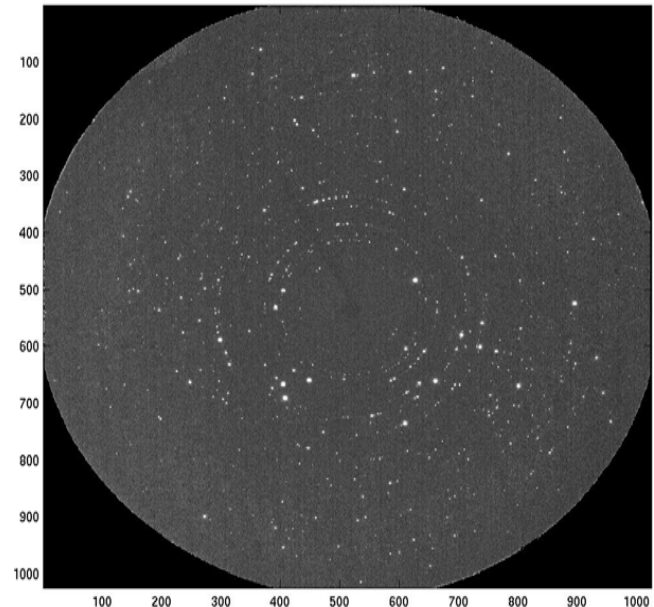
- Oscillation images ($\Delta\omega = 1^\circ$) over extended ω range ($\pm 60^\circ$)
- Data acquisition rate detector limited



- Scan over Euler space



Al_2O_3



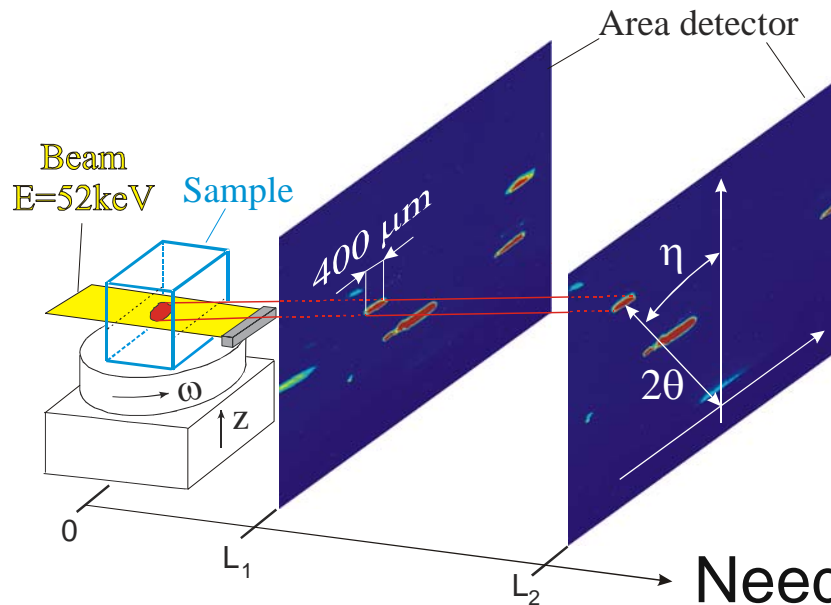
- Few minutes computing time, independent of no. of reflections
- Limit is set by spot overlap – some 1000 grains

E.M. Lauridsen, S. Schmidt, R.M. Suter, H.F. Poulsen, *J. Appl. Cryst.* **34** (2001), 751

Diffraction Tracking

H.F. Poulsen *et al.*, J. Applied Cryst., 2001
R. Suter *et al.*

- Grain position, grain boundary topology
- Crystallographic phase & orientation



- Line focus
- Reflections by ω -rotation
- Projects grain cross section onto detector
- Backtracking \Rightarrow grain outline
- Grain orientation
- Some minutes per layer
- Limitation: mosaic spread

- Grain growth
- Phase transformation
- Initial state before processing

Need to improve:

Flux (emittance, IDs)

Focusing (optics)

Precision Instrumentation

Detectors

Software

High Energy Optics with APS Upgrade

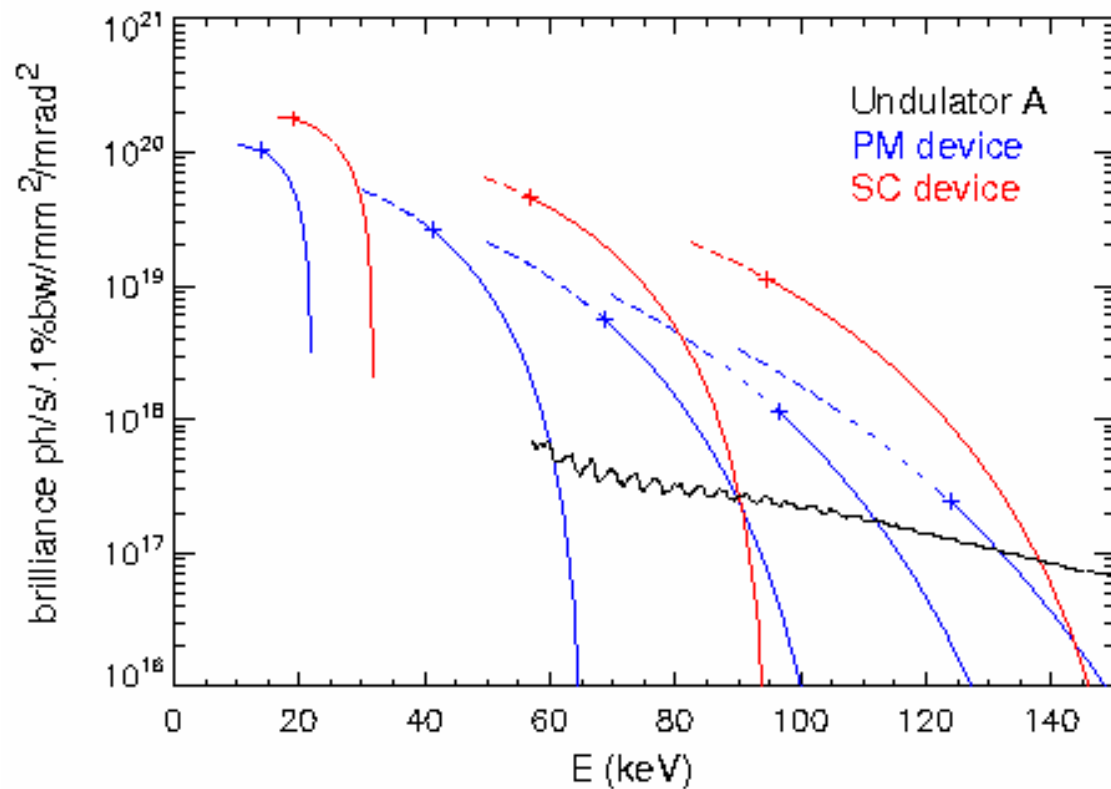
From Shastri

Flux density gains

High Energy Undulator	4 - 10	Permanent magnet device
	Up to 40	Superconducting device
Emittance	6 - 7	High-demag, 2D focusing
	3	High-demag, 1D focusing
	2	No focusing
Ring current	2	
Longer IDs	?	

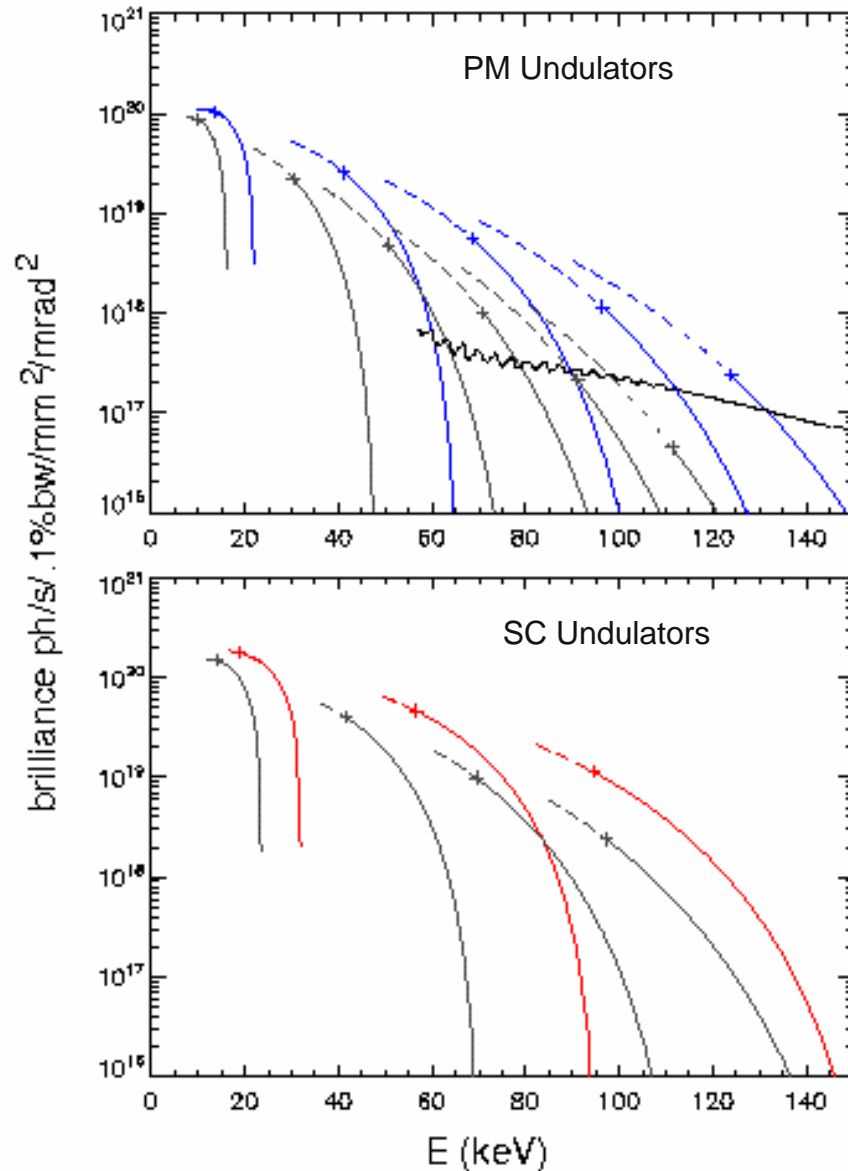
Optimized High-Energy Undulators

(at present emittances *)



	Kmax	Min. gap (mm)	Period (cm)	Periods
UA	2.	11	3.3	70
PM	1.1 (1.5)	8.5 (7)	2.1	114
SC	1.2 (1.4)	8 (7)	1.45	165

7 GeV versus 6 GeV



Blue 7 GeV
Black 6 GeV

It's a big deal

Red 7 GeV
Black 6 GeV

Implication for High-Energy X-ray Science with ERLs, etc.

?

Other Upgrade Issues to Consider

■ Conventional Facilities

- HVAC at the APS is not where it should be
 - *Walk into the 431 lobby for proof*
- Temperature control on floor, in stations, not adequate for extreme precision being proposed

■ Office Space

- LOMs are crowded and cannot house the desired number of staff
- Lack of storage space is recurrent issue

■ Computer Infrastructure

- Lack of computer support is critical, cause of much inefficiency

Conclusions

- APS, ESRF, Spring-8, and in the future Petra-3 are superb sources of high-energy x-rays.
 - NSLS-2, SSRL, Diamond, SLS, etc. are not.
- The high-energy program at the APS is active in many areas
 - PDF
 - 3DXRD
 - WAXS for many applications
 - Diffuse Scattering
 - HESAXS

All will benefit from better undulators, lower emittance, better detectors, optimized beamlines

- Strong movement in the direction of kinetic, *in situ* experiments
- Concerns by many speakers about staffing levels and support for non-expert users
- Huge opportunities for improvement in detectors
- Need to dedicate 11-ID to high-energies
- Possible expansion of high-energy facilities
 - Maybe HP/HE beamline

Future Scientific Areas for the APS

Which is more important?

